

PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION  
International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification <sup>4</sup> : A23C 9/00, 23/00, A23L 1/42 C09K 3/30	A1	(11) International Publication Number: WO 88/ 04896 (43) International Publication Date: 14 July 1988 (14.07.88)
<p>(21) International Application Number: PCT/AU88/00003</p> <p>(22) International Filing Date: 5 January 1988 (05.01.88)</p> <p>(31) Priority Application Number: PH 9795</p> <p>(32) Priority Date: 8 January 1987 (08.01.87)</p> <p>(33) Priority Country: AU</p> <p>(71) Applicant: SOLTEC RESEARCH PTY. LTD. [AU/AU]; 7 Brand Road, Knoxfield, VIC 3180 (AU).</p> <p>(72) Inventors: HALLS, Neil, Graham ; 30 Stableford Avenue, Glen Waverley, VIC 3150 (AU). TOMLINSON, Roderick, Peter ; 32 Highwood Drive, Glen Waverley, VIC 3150 (AU).</p> <p>(74) Agent: EDWD. WATERS &amp; SONS; 50 Queen Street, Melbourne, VIC 3000 (AU).</p>	<p>(81) Designated States: AT (European patent), AU, BE (European patent), CH (European patent), DE (European patent), FR (European patent), GB (European patent), IT (European patent), JP, LU (European patent), NL (European patent), SE (European patent).</p> <p><b>Published</b> <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i></p>	
<p>(54) Title: AEROSOL PRODUCTS</p> <p>(57) Abstract</p> <p>An aerosol dispensable composition comprising in combination: (i) 0.05 to 5 % w/w of a frothing agent; (ii) 0.05 to 75 % w/w of a sweetener; (iii) 0.05 to 10 % w/w of a thickener; (iv) 0.01 to 1 % w/w of a preservative; (v) 0.1 to 10 % w/w a phosphate buffer system adapted to maintain the pH of the compositions in the range 8.0 to 8.5; and the balance being selected from other excipients, flavours and colourants, propellants and water.</p>		

BEST AVAILABLE COPY

*FOR THE PURPOSES OF INFORMATION ONLY*

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	FR	France	ML	Mali
AU	Australia	GA	Gabon	MR	Mauritania
BB	Barbados	GB	United Kingdom	MW	Malawi
BE	Belgium	HU	Hungary	NL	Netherlands
BG	Bulgaria	IT	Italy	NO	Norway
BJ	Benin	JP	Japan	RO	Romania
BR	Brazil	KP	Democratic People's Republic of Korea	SD	Sudan
CF	Central African Republic	KR	Republic of Korea	SE	Sweden
CG	Congo	LI	Liechtenstein	SN	Senegal
CH	Switzerland	LK	Sri Lanka	SU	Soviet Union
CM	Cameroon	LU	Luxembourg	TD	Chad
DE	Germany, Federal Republic of	MC	Monaco	TG	Togo
DK	Denmark	MG	Madagascar	US	United States of America
FI	Finland				

-1-

AEROSOL PRODUCTSFIELD OF THE INVENTION

This invention relates to a composition for use in forming milk shake type products.

5 This invention was conceived as a means for preparing a flavoured thickened foamy milk-based drink as an alternative to the traditional milk shake which was formed by blending milk with syrup powdered flavours and ice-cream in a mechanical blender. It is an object of the invention to provide a similar milk drink using an aerosol dispenser.

10 The requirements of a product of this type should include

(i) compatibility with existing aerosol packaging types in order to minimise manufacturing cost.

15 (ii) a shelf life in an unrefrigerated condition of at least twelve months.

(iii) full compliance with food regulations.

(iv) consumer acceptable flavours as attractive as possible.

20 (v) convenience in use.

(vi) capability of being thickened and frothed in addition to being flavoured.

In order to achieve these objects, it was realised that many constraints existed preventing the formulation of a successful product as discussed below.

25 Most flavours have a limited shelf life in solution due to oxidation, microbial attack, enzymatic breakdown, etc.

30 The product should be of a low viscosity initially, so that it can be ejected rapidly from the aerosol can to provide the necessary mixing action. This is not easily achieved because the product needs to be concentrated and the thickeners and viscosity builders required in the composition are also concentrated.

35 Conventional aerosol cans are not produced to food quality specifications and the lining systems are not able to hold pressurized, low pH liquids for adequate periods of

-2-

time. However, most food products are packed in liquids at a low pH in order to avoid microbial spoilage.

The necessity to provide preservation of the product in the absence of low pH compositions must be achieved from the narrow range of food approved added  
5 preservatives.

The aerosol propellant is normally restricted to noncondensable gases by food regulations in most countries.

Finally food colours are also affected by some of the above parameters and must be properly regulated.  
10

From the above it can be seen that a large number of difficulties stand in the way of providing an acceptable aerosol product.

In PCT/AU/85/00135 an aerosol dispensable composition is disclosed comprising in combination  
15

- (i) 0.05 to 5% of a frothing agent,
- (ii) 0.05 to 75% of a sweetener,
- (iii) 0.05 to 10% of a thickener,
- (iv) 0.01 to 1% of a preservative, and

the balance being other excipients, flavours and colourants, propellants and water. High levels of sweetener are exemplified and believed necessary to maintain bacterial control. This document also discloses the need to maintain the pH in the neutral range, however, the systems disclosed  
20 have failed to eliminate pinhole corrosion within relatively short times e.g. less than six months.  
25

A similar approach is disclosed in U.S. Patent 3,622,354 which relates to a product utilizing high sugar levels to achieve bacterial control and also reveals the use of preservatives at low pH (5-5.5).  
30

In both instances the product produced is not acceptable primarily due to short shelf life and corrosion. Accordingly further investigation has taken place concerning stabilizing such mixtures.

#### 35 SUMMARY OF INVENTION

Surprisingly, a particular phosphate buffer system has been discovered which has been found to effectively

-3-

stabilize the mixture by maintaining pH in the range 8.0 to 8.5.

Accordingly, there is provided an aerosol dispensable composition comprising in combination :

- 5 (i) 0.05 to 5% w/w of a frothing agent;  
(ii) 0.05 to 75% w/w of a sweetener;  
(iii) 0.05 to 10% w/w of a thickener;  
(iv) 0.01 to 1% w/w of a preservative;  
(v) 0.1 to 10% w/w a phosphate buffer system adapted to  
10 maintain the pH of the compositions in the range 8.0 to 8.5;  
and the balance being selected from other excipients,  
flavours and colourants, propellants and water. Preferably,  
the phosphate buffer system is present in an amount ranging  
from 0.1-2.5% w/w. The phosphate buffer system typically  
15 comprises a mixture of potassium phosphate and sodium  
phosphate. This system also allows various types of flavour  
other than chocolate and strawberry to be also practically  
incorporated. More particularly the buffer system can  
comprise a mixture of potassium dihydrogen phosphate and  
20 disodium hydrogen phosphate, the ratio of  $\text{KH}_2\text{PO}_4$  to  $\text{Na}_2\text{HPO}_4$   
being from 1:60 to 1 to 105.

Testing has shown that a composition of the type disclosed in PCT/AU/85/00135 can be stabilized and no corrosion has been detected either at room temperature or under accelerated high temperature storage conditions.  
25 Thus, the product exhibits very long shelf life.

Preferably, the phosphates are blended in the following ratio to maintain the composition pH in the range 8.0-8.5.

- 30  $\text{KH}_2\text{PO}_4$  pH = 4.62 as 1% aqueous solution.  
 $\text{Na}_2\text{HPO}_4$  pH = 9.12 as 1% aqueous solution.  
 $\text{KH}_2\text{PO}_4 : \text{NaHPO}_4 :: 1.0 : 87.4$

The preferred ingredients for the composition of this invention are as follows:

- 35 A. Frothing agent is selected from emulsifiers of the sorbitol ester, glycerol ester or fatty acid sucrose ester types. Most preferred are glycerol monostearate, sorbitol

-4-

monostearate which generally produce the best foam with the least affect on flavour. The frothing agent also aids in the stable dispersion of the flavour agents.

B. The sweetener may be selected from artificial  
5 sweeteners such as saccharine, aspartame, dextrose, fructose or from natural sweeteners such as sucrose. Typically artificial sweeteners are used in the range 0.1 to 5% whilst natural sweeteners are normally in the range 5 to 75%. The most reliable sweetener is sucrose and the level of  
10 sweetener required is constrained by adequacy in providing sweetening effect at the lower end of the scale and by viscosity and solubility at the upper end of the concentration range. Excessive levels when combined with other excipients can produce crystalline growth in the  
15 aerosol cans which results in valve blockages. The most preferred sweetener is sucrose used at a level of between 15% - 75%. Typically, a representative formula is

	%
Flavour	3.0
20 Sucrose	61.0
Water	28.99
AZO Red Dye	0.010
Nitrous Oxide	7.0

25 C. Careful selection of the thickener is required because the product is normally used at a dilution of around 10% and with conventional thickeners such as pectin, tragacanth, the ten-fold concentration in the aerosol product would result in a viscosity that was too high to  
30 allow proper dispensing from the aerosol can. Thus, preferred thickeners are gums of polysaccharide type particularly those of a grade which undergo interaction with milk to produce a large increase in milk viscosity after contact. A number of the gums tested showed a tendency to  
35 age thicken when stored over a period of time. This aging tendency was reduced by addition of water softening components of the phosphate type. Preferred thickeners are

-5-

carragheenan, methylcellulose, hydroxypropylcellulose, and general polysaccharides.

An example of thickeners follows:-

	%
5 Flavour	3.0
Sucrose	50.0
Water	45.95
Xanthane Gum	1.0
Sodium Hexametaphosphate	0.05
10 Nitrous Oxide	7.0

D. The colouring agents should be present in the range of 0.01 to 5% preferably 0.01 to 1%. Variation of the other components of the composition tends to produce variable affects on colour. Many colours also increase the corrosivity of the formula to the aerosol container. All approved dystuff are suitable. The preferred colouring agents are azodyes of the ponceau 4R, erythroscine, carmoisine, tartrazine type, brilliant blue F.C.F., Brown HT, allura red and a wide range of natural colouring substances.

E. Typical examples of the buffer system are as follows:

25	Buffer No. 1:	Dipotassium Dihydrogen Phosphate	77.2% W/W
		Potassium Dihydrogen Phosphate	22.7% W/W
		pH of above combination in water at 2.2% W/W is 8.0	
30	Buffer No. 2:	Potassium Dihydrogen Phosphate	79.0% W/W
		Sodium Hydroxide	21.0% W/W
		pH of above combination in water at 0.7% is 8.1%	
35	Buffer No. 3:	Sodium Dihydrogen Phosphate	4.3% W/W
		Disodium Hydrogen Phosphate	95.7%
		pH of above combination in water at 6.8% is 8.0	

Each buffer introduces stability and performance to the dispensable composition described. This system ensures

-6-

microbiostatic, microbiocidal and fungicidal affect and as well reduce corrosion of lacquered tinplate to the point where shelf life if in excess of twelve months at 20°C are now achievable.

5 F. The balance of the mixture is made up with purified water.

G. The propellant to be used to dispense the composition from the aerosol can well comprise 2 to 50% by weight and can comprise any food approved propellant.  
 10 Preferably nitrous oxide, carbon dioxide and octafluorocyclobutane, or propane may be used. However, the most preferred propellant is nitrous oxide in order to achieve the optimum performance required for this particular composition. Due to its non-condensable nature a higher  
 15 pressure can be used to ensure mixing with nitrous oxide than with condensable propellants. In addition, the condensable propellants tend to produce a shaving-cream type foam due to their densities and settle out of the system. Care must be taken if carbon dioxide is used as propellant  
 20 because it is acidic. However, it could be used where lined aerosol cans are used.

#### EXAMPLES

##### EXAMPLE 1

	<u>COMPONENT</u>	<u>% W/W</u>
25	Flavour	0.1 - 3.0
	Sodium Benzoate	0.01 - 1.0
	Polysaccharide Gum	0.01 - 1.0
	Dye Stuff	0.001 - 0.1
	Glyceryl Monostearate	0.01 - 1.0
30	Nitrous Oxide	0.5 - 6.0
	Sucrose	20 - 45
	Water	BALANCE
	Butylated Hydroxy Anisole	0.001 - 0.5
	Disodium Hydrogen Phosphate	0.01 - 2.0
35	Potassium Dihydrogen Orthophosphate	0.01 - 0.5



-7-

EXAMPLE 2

	<u>COMPONENT</u>	<u>%</u>
	Flavour	3.000
	Sucrose	50.000
5	Sorbitol ester	2.000
	Hydroxy propyl cellulose	0.080
	Buffer No. 1 (aforestated)	2.200
	Colour	0.008
	Water	TO 200%

10 EXAMPLE 3

	Flavour	3.000
	Sucrose	50.000
	Sorbitol ester	1.000
	Hydroxy propyl cellulose	0.080
15	Buffer No. 2 (aforestated)	0.700
	Colour	0.008
	Water	TO 100%

EXAMPLE 4

	Flavour	3.000
20	Sucrose	50.000
	Sorbitol ester	1.000
	Hydroxy propyl cellulose	0.080
	Buffer 3 (aforestated)	6.800
	Colour	0.008
25	Water	TO 100%

In each of the examples, testing over a prolonged period of months has failed to reveal any corrosion spoilage or bacterial spoilage.

Aerosol Milk shake compositions utilizing the buffer system achieve a long life and permit a wider range of flavours than previously possible.

35

-8-

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:

1. An aerosol dispensable composition comprising in combination:
  - (i) 0.05 to 5% w/w of a frothing agent;
  - (ii) 0.05 to 75% w/w of a sweetener;
  - (iii) 0.05 to 10% w/w of a thickener;
  - (iv) 0.01 to 1% w/w of a preservative;
  - (v) 0.1 to 10% w/w a phosphate buffer system adapted to maintain the pH of the compositions in the range 8.0 to 8.5; and the balance being selected from other excipients, flavours and colourants, propellants and water.
2. The composition according to Claim 1 wherein the phosphate buffer system is present in an amount ranging from 0.1 to 2.5% w/w.
3. The composition according to Claim 1 wherein the phosphate buffer system comprises a mixture of potassium phosphates and sodium phosphates.
4. The composition according to Claim 1 wherein the phosphate buffer system comprises a mixture of disodium hydrogen phosphate and potassium dihydrogen orthophosphate.
5. The composition according to Claim 1 wherein the phosphate buffer system comprises a mixture of potassium dihydrogen phosphate and disodium hydrogen phosphate, the ratio of  $\text{KH}_2\text{PO}_4$  to  $\text{Na}_2\text{HPO}_4$  being from 1:60 to 1 to 105.
6. An aerosol dispensable composition according to Claim 1 wherein the frothing agent is selected from emulsifiers of the sorbitol ester, glycerol ester and fatty acid sucrose ester types.

-9-

7. An aerosol dispensable composition according to Claim 1 wherein the frothing agent is glycerol monostearate and/or sorbitol monostearate.

8. An aerosol dispensable composition according to Claim 1 wherein the sweetener is selected from the group of artificial sweeteners including saccharine, aspartame, dextrose and fructose.

9. An aerosol dispensable composition according to Claim 1 wherein the sweetener is an artificial sweetener present in amounts from 0.1 to 5%.

10. An aerosol dispensable composition according to Claim 1 wherein the sweetener is a natural sweetener present in amounts of from 5 to 75%.

11. An aerosol dispensable composition according to Claim 1 wherein the thickener is selected from the group of gums of the polysaccharide type including carragheenan, methylcellulose, hydroxypropylcellulose and general polysaccharides.

12. An aerosol dispensable composition according to Claim 1 wherein the colourants are present in the range of 0.01 to 5%.

13. An aerosol dispensable composition according to Claim 1 wherein the colourants are selected from the group of asodyes of the ponceau 4R, erythroscine, carmoisine, tartrazine type, brillian blue F.C.F., brown HT and allura red.

-10-

14. An aerosol dispensable composition according to Claim 1 wherein the propellant is selected from nitrous oxide, carbon dioxide, octafluorocyclobutane and propane.

# INTERNATIONAL SEARCH REPORT

International Application No PCT/AU 88/00003

<b>I. CLASSIFICATION OF SUBJECT MATTER</b> (1) several classification symbols apply, indicate all (2) According to International Patent Classification (IPC) or to both National Classification and IPC		
Int. Cl. <sup>4</sup> A23C 9/00, 23/00, A23L 1/42, C09K 3/30		
<b>II. FIELDS SEARCHED</b>		
Minimum Documentation Searched (3)		
Classification System	Classification Symbols	
IPC US Cl.	A23C 9/00, 23/00, A23L 1/19, 1/34, 1/42, C09K 3/30 426/317	
Documentation Searched other than Minimum Documentation to the extent that such documents are included in the fields searched (4)		
AU : IPC as above		
<b>III. DOCUMENTS CONSIDERED TO BE RELEVANT (5)</b>		
Category (6)	Citation of Document, (7) with indication, where appropriate, of the relevant passages (8)	Relevant to Claim No. (9)
X	WO,A, 86/00196 (SOLTEC RESEARCH PTY. LTD, TOMLINSON) 16 January 1986 (16.01.86) See page 5, lines 18-27, especially line 25; and claims.	(1-14)
X	US,A, 3622354 (DIAMOND) 23 November 1971 (23.11.71) See especially column 1, lines 15-20; column 3, lines 32-34; examples 4-8; and claims.	(1,2,6-14)
X	US,A, 4343825 (TAKEDA et al) 10 August 1982 (10.08.82) See column 2, lines 29-35.	(1-14)
X	AU,B, 28005/71 (453541) (CARNATION COMPANY) 26 October 1972 (26.10.72) See especially page 15, example 1 and page 8, lines 2-10.	(1-14)
A	EP,A, 135768 (ASAHI DENKA KOGYO KABUSHIKI KAISHA) 3 April 1985 (03.04.85) See page 8, lines 20-21; page 7, lines 10-14; page 9, line 16; and page 9, line 22.	
Y	US,A, 4571338 (OKONOGI et al) 18 February 1986 (18.02.86) See especially column 2, lines 24-31 and column 3, lines 29-33.	(1-13)
Y	GB,A, 1449271 (HERRING et al) 15 September 1976 (15.09.76) See column 1, lines 9-12	(14)
(continued)		
<div style="display: flex; justify-content: space-between;"> <div style="width: 45%;"> <p>* Special categories of cited documents: (10)</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 50%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"A" document member of the same patent family</p> </div> </div>		
<b>IV. CERTIFICATION</b>		
Date of the Actual Completion of the International Search 27 April 1988 (27.04.88)	Date of Mailing of this International Search Report (03.05.88) 3 MAY 1988	
International Searching Authority Australian Patent Office	Signature of Authorized Officer (R.E. GRANT)	

## FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET

- A R.C. WEAST, "CRC Handbook of Chemistry and Physics",  
66th edition, published 1985, by CRC Press, Inc.  
(Boca Raton, Florida), See page D-144.

## VI. OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1. ☐ Claim numbers . . . . . because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claim numbers . . . . . because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claim numbers . . . . . because they are dependent claims and are not drafted in accordance with the second and third sentences of PCT Rule 6.4(a).

## VII. OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING

This International Searching Authority found multiple inventions in this international application as follows:

1. ☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4. ☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

## Remarks on Protest

- ☐ The additional search fees were accompanied by applicant's protest.  
☐ No protest accompanied the payment of additional search fees.

ANNEX TO THE INTERNATIONAL SEARCH REPORT ON  
INTERNATIONAL APPLICATION NO. PCT/AU 88/00003

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report		Patent Family Members			
WO	8600196	AU	44995/85	EP	190158
US	3622354				
US	4343825	JP	56042555		
AU	28005/71	CA	919493	DE	2122033
		GB	1299304	IT	1004011
				FR	2091171
				US	3851071
EP	135768	JP	60064626		
US	4571338	DE	3425284	JP	60019452
GB	1449271	CA	1006747	IE	39174

END OF ANNEX

**THIS PAGE BLANK (USPTO)**



**This Page is Inserted by IFW Indexing and Scanning  
Operations and is not part of the Official Record**

**BEST AVAILABLE IMAGES**

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images include but are not limited to the items checked:

- ☒ **BLACK BORDERS**
- ☒ **IMAGE CUT OFF AT TOP, BOTTOM OR SIDES**
- ☐ **FADED TEXT OR DRAWING**
- ☐ **BLURRED OR ILLEGIBLE TEXT OR DRAWING**
- ☐ **SKEWED/SLANTED IMAGES**
- ☐ **COLOR OR BLACK AND WHITE PHOTOGRAPHS**
- ☐ **GRAY SCALE DOCUMENTS**
- ☐ **LINES OR MARKS ON ORIGINAL DOCUMENT**
- ☐ **REFERENCE(S) OR EXHIBIT(S) SUBMITTED ARE POOR QUALITY**
- ☐ **OTHER:** \_\_\_\_\_

**IMAGES ARE BEST AVAILABLE COPY.**

**As rescanning these documents will not correct the image problems checked, please do not report these problems to the IFW Image Problem Mailbox.**

**THIS PAGE BLANK (USPTO)**